Letters

The effect of heat on ion-implanted glass-supported tantalum thin films

Tantalum thin films are widely used as resistors in hybrid integrated circuits because of their excellent stability, low thermal coefficient of resistivity and wide variation of resistivity with impurities. In the University of Surrey work has been carried out to control the electrical properties of ion implanted tantalum films. These films are usually sputtered on glass, which has good surface properties and is inexpensive.

During the preparation, implantation and use of tantalum films, they may be considerably heated, affecting their physical properties. Thus it was considered worthwhile to study the crystallographic structure of tantalum films after heat-treatment. The best method of studying changes in the structure of materials that are likely to affect the electrical properties, such as crystal size and dislocation density, is X-ray line broadening. The experimental problems involved in producing good quality X-ray diffraction profiles for line-broadening studies from the glass-supported thin films proved to be insuperable, so an examination was carried out on ball-milled tantalum powder to obtain the annealing behaviour of the bulk material. This procedure was used successfully previously for studies of ion-implantation and mechanical surface damage in gallium arsenide [1], where similar problems were encountered.

Powdered tantalum was obtained by carefully filing a sheet of pure tantalum and screening the filings. The specimens were annealed in evacuated quartz tubes at temperatures ranging from 400 to 1000° C, in steps of 100° C, for 1 h each step. Line-broadening due to the specimen, β^*_s, was obtained by subtracting the instrumental broadening, β^*_I, from the total broadening, β^*_T, using the equation [2]

$$\beta^*_s = \beta^*_T - \frac{(\beta^*_I)^2}{\beta^*_T} \qquad (1)$$

where $\beta^* = \beta\cos\theta/\lambda$; β = integral breadth; θ = Bragg angle; λ = wavelength of X-rays.

The instrumental broadening here (β^*_I) was obtained from the 1000° C annealed tantalum. Values of microstrain, E, and crystallite size, D, were obtained from the equation [2]

$$(\beta^*_s)^2 = \frac{1}{D^2} + \frac{16E^2\sin^2\theta}{\lambda^2}. \qquad (2)$$

The plot of $(\beta^*_s)^2$ against $(d^*)^2 = (2\sin\theta/\lambda)^2$ gave the values of D and E. $d^* = 1/d$; d = interplanar spacing. The variation of microstrain and crystallite size with the annealing temperatures is shown in Fig. 1. During the measurement of particle size and microstrain, all the X-ray diffraction profiles were observed to become displaced with increasing annealing temperatures, indicating that the d spacing was increasing (Fig. 2). A similar effect has been observed by Schoening [3] while annealing tantalum in hydrogen, and attributed to absorbed gas.

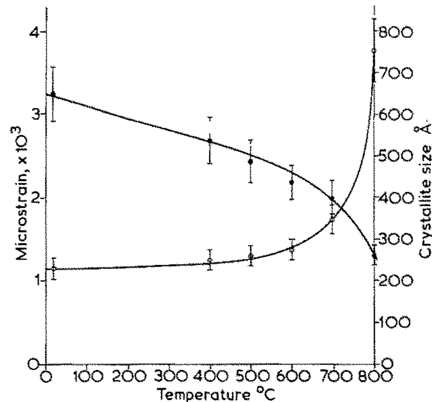

Figure 1 Effect of annealing temperatures on crystallite size and microstrain in ball milled tantalum. ○ Crystallite size, ● microstrain.

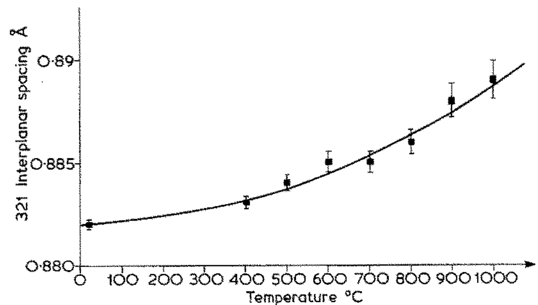

Figure 2 Shift of the tantalum (321) peak due to heat-treatment for 1 h.

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Thin films of tantalum were obtained by sputtering tantalum onto glass in a vacuum of 10^{-6} Torr. The thickness of the film was about 500 Å, with area 25 mm x 25 mm. To obtain implanted material, tantalum films of 500 Å thickness were implanted with nitrogen ions of energy 40 keV and a dose of 1.6×10^4 ions cm^{-2}. Strips 1 mm wide were cut from unimplanted and nitrogen implanted tantalum films. They were mounted in Philips powder cameras, 11.46 cm diameter. $CuK\alpha$ radiation with a nickel filter was used. The specimens were not rotated. The specimens were then annealed in evacuated quartz tubes at 400, 500 and 600° C for 1 h. X-ray powder photographs were taken after each anneal.

The d spacings of the reflection lines from the unannealed specimens corresponded to those of b c c tantalum and β-tantalum. Both phases showed preferred orientation. After annealing at temperatures of 400 and 500° C, the X-ray diffraction patterns showed little change from those of the as-sputtered tantalum. Annealing at 600° C caused the rings from b c c tantalum and β-tantalum to disappear. The diffraction pattern of the implanted unannealed specimen showed some continous rings from TaN. Preferred orientation of the β-tantalum was observed but not of the b c c tantalum. Annealing at 600° C caused the TaN rings to sharpen, indicating that it had recrystallized.

The dislocation density for b c c crystals can be calculated from the equation given by Williamson and Smallman [4] as follows:

$$\rho = 14.4 \frac{E^2}{b^2}$$

where ρ = dislocation density; E = strain value; b = Burgers vector.

It can be seen from Fig. 1 that the microstrain dropped by about 25% on heating from room temperature to 600° C, thus the dislocation density, which varies as the $(\text{strain})^2$, will drop by over 40% with a corresponding effect on the resistivity.

The formation of the β-tantalum in the sputtered tantalum films is in agreement with the observations of Read and Altman [5]. They found that β-tantalum is unstable, changing into b c c tantalum at ~700° C when heated in a vacuum of 10^{-8} Torr.

It can thus be seen that heat produces many complex structural changes in ion-implanted glass supported tantalum films. It may be assumed that most of these changes will produce corresponding changes in the electrical properties of the films.

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A relationship between undercooling and atomized powder diameter

An expression between undercooling ΔT, and atomized powder diameter D can be derived if we consider that when continuous cooling occurs the undercooling will be [1]

$$\Delta T^2 = k(\dot{T}) \tag{1}$$

in which k is a constant depending on the system being solidified and $\dot{T}$ is the cooling rate. The above equation should be valid for any composition invariant transformation in which the nucleation of the product phase can be described by classical nucleation theory [1]. Equation 1 applies to nucleation during continuous cooling and we apply it here to undercooled atomized particles experiencing a high cooling rate, assuming that the solidification process immediately after nucleation is composition invariant. Assuming that a sphere contains negligible temperature gradients, the cooling rate $\dot{T}$ for convection and radiation cooling will be given by

$$\dot{T} = \frac{6}{D\rho C_p}[h_c(T-T) + \epsilon\sigma(T^4 - T_0^4)]. \tag{2}$$

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